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(54) PROPYLENE OXIDE PURIFICATION

REINDARSTELLUNG VON PROPYLENOXID

PURIFICATION D'OXYDE DE PROPYLENE

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(73) Proprietor: **ARCO Chemical Technology, L.P.**
Greenville, Delaware 19807 (US)

(72) Inventor: **RUETER, Michael, A.**
Norristown, PA 19401 (US)

(74) Representative: **Kooy, G.L. et al**
Octrooibureau Vriesendorp & Gaade,
Dr. Kuyperstraat 6
2514 BB The Hague (NL)

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US-A- 3 715 284

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EP 1 003 733 B1

Description

[0001] This invention provides a method of recovering propylene oxide in purified form from an epoxidation reaction mixture additionally comprised of methanol and contaminating amounts of acetaldehyde impurity. Such mixtures may be formed by epoxidizing propylene with hydrogen peroxide using a titanium silicalite as a catalyst and methanol as a reaction solvent. Substantially all of the acetaldehyde may be removed by fractionation of the epoxidation reaction mixture in a distillation column wherein a concentration of methanol is maintained in the distillation column sufficient to suppress the volatility of the acetaldehyde so as to minimize the amount of acetaldehyde present in the overhead stream.

[0002] In recent years, the production of propylene oxide from propylene using hydrogen peroxide as an oxidant and a titanium-containing zeolite as a zeolite as a catalyst has been proposed. Methanol is a particularly preferred reaction solvent for such purposes, as it tends to promote high catalyst activity and selectivity. Epoxidation processes of this type are described for example, in U.S. Pat. Nos. 5,591,875,4,833,260, 5,621,122, 5,646,314, and 4,824,976, EP Pub. No. 0732327, and Clerici et al., J. Catalysis 129, 159-167 (1991). Although such processes are capable of providing exceptionally high selectivity to propylene oxide, minor quantities of certain by-products such as acetaldehyde are inevitably formed. Since a satisfactory propylene oxide for commercial purposes should contain less than 100 ppm, and preferably less than 20 ppm, acetaldehyde, the development of methods for separating substantially all of the acetaldehyde by-product from such reaction mixtures is necessary. In addition, epoxidation processes of this type form water as a co-product with the water being derived from the hydrogen peroxide oxidant. Depending upon the method used to generate the hydrogen peroxide to be used in the epoxidation reaction, water may also be present in the feed to the reactor. While epoxidation processes catalyzed by titanium-containing zeolites are remarkably tolerant of water, it will be necessary for most commercial purposes to obtain propylene oxide in substantially anhydrous form. An efficient method of removing water from the propylene oxide produced by such an epoxidation process therefore is needed.

[0003] Distillative methods for preparing purified propylene oxide are disclosed in US 3715284 and EP 0675119A.

[0004] Prior art methods for separating acetaldehyde from propylene oxide, which were principally developed in connection with organic hydroperoxide-based epoxidation processes wherein either ethylbenzene or t-butyl alcohol is used as a reaction solvent, have generally charged the crude propylene oxide, after removing substantially all the unreacted propylene, to a distillation column and removed propylene oxide and all lower boiling materials, including acetaldehyde, as an overhead product. A subsequent fractionation of the overhead product in a second distillation column then is employed to separate acetaldehyde as an overhead product from a bottoms fraction containing the propylene oxide.

[0005] We have now found that the substantial quantity of methanol present as a reaction solvent in a crude epoxidation product from a hydrogen peroxide/titanium silicalite process may be used to advantage in achieving the desired separation of acetaldehyde from propylene oxide. The presence of methanol at high concentrations substantially reduces the volatility of acetaldehyde relative to propylene oxide. Thus, instead of taking acetaldehyde overhead as in conventional purification schemes, the present invention operates by maintaining a concentration of methanol in the liquid phase within the distillation column sufficiently great so as to suppress the volatility of acetaldehyde and force all or substantially all of the acetaldehyde into a bottoms stream. Purified propylene oxide containing a reduced level of acetaldehyde is taken overhead. The overhead stream will also have a substantially lower water content as compared to that of the crude epoxidation reaction product.

[0006] The present invention provides a method of purifying propylene oxide produced in an epoxidation process wherein methanol is used as a solvent comprising the steps of

(a) feeding a crude epoxidation reaction product comprised of propylene oxide, methanol and acetaldehyde to a fractionator;
(b) subjecting the crude epoxidation reaction product to fractional distillation within the fractionator;
(c) withdrawing an overhead stream comprised of propylene oxide and methanol and having a reduced level of acetaldehyde as compared to the crude epoxidation reaction product from the fractionator; and
(d) withdrawing a bottoms stream comprised of methanol and acetaldehyde from the fractionator.

Said fractional distillation is performed under conditions such that the concentration of methanol within the fractionator is maintained at a level sufficiently high so as to substantially prevent acetaldehyde from being present at the point at which the overhead stream is withdrawn from the fractionator.

[0007] More specifically, the present invention provides a method of purifying a crude epoxidation reaction product comprised of 2 to 10 weight percent propylene oxide, 60 to 85 percent methanol, 10 to 25 percent water and 0.01 to 0.1 weight percent acetaldehyde comprising the steps of

(a) feeding the crude epoxidation reaction product to a fractionator having from 20 to 60 theoretical vapour-liquid

contacting stages;

(b) subjecting the crude epoxidation reaction product to fractional distillation within the fractionator at a top pressure of 15 to 50 psia (103.4 to 344.7 kPa), a bottoms temperature of 80°C to 110°C, and a reflux:distillate ratio of 10:1 to 30:1;

(c) withdrawing an overhead stream comprised of propylene oxide and at least 2 weight percent methanol from the fractionator; and

(d) withdrawing a first bottoms stream comprised of methanol, water and at least 99% of the acetaldehyde present in the crude epoxidation reaction product from the fractionator.

DESCRIPTION OF THE DRAWING

[0008] The accompanying drawing (Figure 1) illustrates in schematic form a particular embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

[0009] A crude epoxidation reaction product, from which unreacted propylene has been substantially removed by prior distillation such as a flash distillation or other such conventional distillation operation, is fed through line 2 to an intermediate zone of first fractionator 1. Depending upon the epoxidation conditions employed, the crude epoxidation reaction product generally has a composition comprised of the following components, in percent by weight:

propylene oxide	2-10
methanol	60-85
acetaldehyde	0.01-0.1
water	10-25
ring-opened byproducts of propylene oxide (e.g., propylene glycol)	0.1-1
propylene and/or propane	0.01-0.1

[0010] The first fractionator 1 may comprise a conventional distillation column or tower having a capacity appropriate to handle the desired volume of crude epoxidation reactor product within a given period of time and having a sufficient number of theoretical plates to accomplish the necessary separation of methanol and acetaldehyde from the propylene oxide. The use of a plate column is particularly advantageous. For economic reasons, the fractionator should normally comprise a single distillation column or tower, although the use of multiple columns or towers to accomplish the same result is not excluded.

[0011] Fractionator 1 is operated at conditions such that a first overhead stream comprised of propylene oxide and a minor amount of methanol can be removed through line 3 and substantially all of the acetaldehyde (preferably, at least 99%, more preferably, at least 99.9%) is removed in a first bottoms stream through line 4. It is essential that such conditions be selected to provide a concentration of methanol within the first fractionator 1 which is effective to render the acetaldehyde less volatile than the propylene oxide. Enough methanol must be taken overhead from the first fractionator such that a zone exists above the feed tray where both methanol and propylene oxide concentrations are sufficiently high for acetaldehyde to be heavier (i.e., less volatile) than both methanol and propylene oxide. This will prevent acetaldehyde from reaching the top of first fractionator 1 and permits, under optimum conditions, essentially all of the acetaldehyde in the feed to the first fractionator to be recovered in the bottoms stream. Typically, the process is operated so as to have at least 2 (more preferably, at least 4) weight percent methanol in the overhead stream. To minimize the extent of additional downstream processing which is used, first fractionator 1 is preferably operated at a top pressure of from about 15 to about 50 psia (103.4 to 344.7 kPa) and at a bottoms temperature of from about 80°C to about 110°C. It is particularly advantageous to operate first fractionator 1 such that the top zone thereof is at a pressure of about 25 to 40 psia (172.4 to 275.8 kPa) and the bottom zone thereof is at a temperature of about 93°C to 104°C. First fractionator 1 generally contains from 20 to 60 (more preferably, 30 to 50) theoretical vapour-liquid containing stages. A suitable reflux:distillate ratio is important in achieving optimum results; this reflux ratio should preferably range between 10:1 and 30:1 (more preferably, 15:1 to 25:1). Heat to the fractionator may be supplied by means of a reboiler.

[0012] The first bottoms stream typically will contain substantial amounts of methanol and water in addition to the acetaldehyde, together with other minor by-products such as propylene glycol which have volatilities under the distillation conditions less than that of propylene oxide. An additional advantage of the process of this invention is that it is capable of producing propylene oxide which is substantially free of water, as the bottoms stream will typically contain nearly all (e.g., at least 99%) of the water originally present in the crude epoxidation reaction product. The bottoms stream may be purified by conventional means such as fractional or extractive distillation to recover the methanol for

reuse as the solvent in the epoxidation process.

[0013] Where significant amounts of propylene remain the first overhead stream, which typically will also contain about 90 to 98 weight percent propylene oxide, 2 to 6 weight percent methanol, 0 to 2 weight percent propylene, and less than 20 ppm (more preferably, less than 5 ppm) acetaldehyde, a second fractionator may be utilized to remove said residual propylene. The first overhead stream thus may be charged to an intermediate zone of second fractionator 5 through line 3. Fractionator 5 which is operated so that the propylene is withdrawn as a second overhead stream from a top zone through line 6. Propylene oxide which is essentially free of propylene is withdrawn as a second bottoms fraction from a bottom zone of second fractionator 5 through line 7. Second fractionator 5 is operated advantageously at a top zone pressure of from about 140 to about 300 psia (965.3 to 2068.4 kPa) and at a bottom zone temperature of from about 115°C to about 150°C. Particularly preferred operation involved a pressure of about 160 to 220 psia (1103.2 to 1516.8 kPa) in the top zone thereof and a temperature of about 120°C to 140°C in the bottom zone thereof. From about 5 to about 15 (more preferably, 5 to 10) theoretical vapour-liquid contacting stages are preferably present in the second fractionator. The reflux to distillate ratio is suitably from 10:1 to 50:1 with 20:1 to 40:1 being the preferred range.

[0014] Further purification of the propylene oxide present in the second bottoms stream may be accomplished if so desired by subjecting said stream to extractive distillation. Extractive distillation of impure propylene oxide fractions is well known in the art and is described, for example, in extensive detail in the following U.S. patents: 3,337,425, 3,338,800, 3,464,897, 3,578,568, 3,843,488, 4,140.588, 4,971,661, 5,000,825, 5,006,206, 5,116,465, 5,116,466, 5,116,467, 5,129,996, 5,133,839, 5,139,622, 5,145,561, 5,145,563, 5,154,803, 5,154, 804, 5,160,587, 5,340,446, 5,453,160, 5,464,505 and 5,620,568.

[0015] For example, the second bottoms stream (or, alternatively, the first overhead stream, particularly where the first overhead stream contains little or no unreacted propylene) is fed through line 7 to an intermediate zone of extractive distillation column 8 wherein it is in countercurrent contact with an extractive solvent such as a hydrocarbon (e.g., octane), heavy polar organic compound (e.g., propylene glycol), or such other substance known in the art to be useful for such purpose. Propylene oxide of high purity is removed from a top zone of column 8 through line 9, while the extractive solvent containing relatively less volatile impurities such as water, methanol and the like are removed as an extract stream from a bottom zone of column 8 through line 10. The extract stream is fed to an intermediate zone of stripper 11, which is so operated that components having boiling points above propylene oxide are removed overhead via line 12. The extractive solvent is removed via line 13 as a bottoms stream for recycle to an upper zone of extractive distillation column 8 via line 14. Make-up extractive solvent can be added to line 14 from line 15.

EXAMPLE

[0016] In a first distillation column containing 40 theoretical stages (including reboiler), a crude epoxidation reaction product from which unreacted propylene is largely removed is introduced at the 16th stage from the top of the column. A total condenser is used so that distillate product is withdrawn as a liquid. The feed to the column is as follows:

Component	Wt. %
Propylene Oxide	7.2
Water	16.9
Methanol	74.8
Acetaldehyde	0.04
Propylene & Propane	0.06
Ring-Opened Products	0.95
Other heavy components	0.05

[0017] The first distillation column is operated at a molar reflux ratio (reflux to distillate) of 19.7. The pressure in the column condenser is set at 30 psia (206.8 kPa) and the column operated with a pressure drop of 0.2 psi (1.4 kPa) per tray, resulting in a bottom pressure of about 38 psia (262 kPa). The bottoms (reboiler) temperature is 98°C and the top (condenser) temperature is 51°C.

[0018] Under these conditions, 99.93% of the acetaldehyde in the feed to the column is recovered in the bottom stream and 99.7% of the propylene oxide is recovered overhead. The compositions of these two product streams are as follows:

Component	Distillate Wt.%	Bottoms Wt.%
Propylene Oxide	94.8	0.023
Water	0.0006	18.2
Methanol	4.4	80.5
Acetaldehyde	0.00035	0.044
Propylene & Propane	0.8	0
Ring-Opened Products	0	1.03
Other Heavy Components	0	0.2

Claims

1. A method of purifying a crude epoxidation reaction product comprised of 2 to 10 weight percent propylene oxide, 60 to 85 percent methanol, 10 to 25 percent water and 0.01 to 0.1 weight percent acetaldehyde comprising the steps of

   (a) feeding the crude epoxidation reaction product to a fractionator having from 20 to 60 theoretical vapour-liquid contacting stages;
   (b) subjecting the crude epoxidation reaction product to fractional distillation within the fractionator at a top pressure of 15 to 50 psia (103.4 to 344.7 kPa), a bottoms temperature of 80°C to 110°C, and a reflux:distillate ratio of 10:1 to 30:1;
   (c) withdrawing an overhead stream comprised of propylene oxide and at least 2 weight percent methanol from the fractionator; and
   (d) withdrawing a first bottoms stream comprised of methanol, water and at least 99% of the acetaldehyde present in the crude epoxidation reaction product from the fractionator.

2. The method of claim **1** wherein said epoxidation process utilizes a titanium containing-zeolite as a catalyst.

3. The method of claim **1** wherein said epoxidation process utilizes hydrogen peroxide as an oxidant.

4. The method of claim **1** wherein the concentration of methanol in the crude epoxidation reaction product is 60 to 80 weight percent.

5. The method of claim **1** wherein the crude epoxidation reaction product initially contains propylene and the crude epoxidation reaction product prior to step (a) is subjected to an initial distillation wherein a substantial portion of the propylene is removed.

6. The method of claim **1** wherein the crude epoxidation reaction product and the bottoms stream are additionally comprised of water.

7. The method of claim **1** wherein the overhead stream is fed to a second fractionator wherein the overhead stream is subjecte, to distillation such that a second overhead stream comprised of propylene and a second bottoms stream comprised of propylene oxide and substantially free of propylene are separately withdrawn from the second fractionator.

8. The method of claim **7** wherein the second bottoms stream is fed to an extractive distillation column wherein the second bottoms stream is subjected to extractive distillation with an extractive solvent such that a third overhead stream consisting essentially of propylene oxide and a third bottoms stream comprised of the extractive solvent and impurities present in the second bottoms stream are separately withdrawn from the extractive distillation column.

9. The method of claim **8** wherein the extractive solvent is selected from the group consisting of propylene glycol and octane.

10. A method of purifying propylene oxide produced in an epoxidation process wherein methanol is used as a solvent, hydrogen peroxide is used as an oxidant, and a titanium-containing zeolite is used as a catalyst comprising the

steps of

(a) feeding a crude epoxidation reaction product comprised of 2 to 10 weight percent propylene oxide, 60 to 85 weight percent methanol, 10 to 25 weight percent water and 0.01 to 0.1 weight percent acetaldehyde to a first fractionator having from 20 to 60 theoretical vapor-liquid contacting stages;
(b) subjecting the crude epoxidation reaction product to fractional distillation within the fractionator at a top pressure of 15 to 50 psia, a bottoms temperature of 80°C to 110°C, and a reflux:distillate ratio of 10:1 to 30:1;
(c) withdrawing an overhead stream comprised of propylene oxide and at least 2 weight percent methanol from the fractionator;
(d) withdrawing a bottoms stream comprised of methanol, water and at least 99% of the acetaldehyde present in the crude epoxidation reaction product from the fractionator.

11. The method of claim **10** wherein the top pressure is 25 to 40 psia (172.4 to 275.8 kPa), the bottoms temperature is from 93°C to 104°C, and the reflux:distillate ratio is from 15:1 to 25:1.

12. The method of claim **11** wherein the overhead stream is comprised of at least 4 weight percent methanol.

13. The method of claim **12** wherein the bottom stream contains at least 99% of the water present originally in the crude epoxidation reaction product.

Patentansprüche

1. Verfahren zum Reinigen eines rohen Epoxidierungsreaktionsprodukts umfassend 2 bis 10 Gewichtsprozent Propylenoxid, 60 bis 85 Prozent Methanol, 10 bis 25 Prozent Wasser und 0,01 bis 0,1 Gewichtsprozent Acetaldehyde, umfassend die Schritte von

a. Führen eines rohen Epoxidierungsreaktionsprodukts zu einer Fraktioniervorrichtung mit 20 bis 60 theoretischen Dampf/Flüssigkeitskontaktschritten;

b. Unterwerfen des rohen Epoxidierungsreaktionsprodukts einer Fraktionierdestillation innerhalb der Fraktioniervorrichtung mit einem Oberdruck von 15 bis 50 psia (103,4 bis 344,7 kPa), einer Bodentemperatur von 80°C bis 110°C und einem Rückfluß/Destillatverhältnis von 10:1 bis 30:1;

c. Zurückziehen von der Fraktioniervorrichtung eines Oberstroms, umfassend Popylenoxid und zumindest 2 Gewichtsprozent Methanol; und

d. Zurückziehen von der Fraktioniervorrichtung eines ersten Unterstroms umfassend Methanol, Wasser und zumindest 99% von dem in dem rohen Epoxidierungsreaktionsprodukt vorhandenen Acetaldehyde.

2. Verfahren nach Anspruch 1, wobei der Epoxidierungsprozeß ein Titan enthaltender Zeolith als Katalysator verwendet.

3. Verfahren nach Anspruch 1, wobei der Epoxidierungsprozeß Hydrogenperoxid als ein Oxidans verwendet.

4. Verfahren nach Anspruch 1, wobei die Konzentration von Methanol in dem rohen Epoxidierungsreaktinsprodukt 60 bis 80 Gewichtsprozent ist.

5. Verfahren nach Anspruch 1, wobei das rohe Epoxidierungsreaktionsprodukt anfänglich Propylen umfaßt und das rohe Epoxidierungsreaktionsprodukt vor Schritt a. einer ersten Destillation unterworfen wird, wobei ein beträchtliches Teil des Propylens entfernt wird.

6. Verfahren nach Anspruch 1, wobei das rohe Epoxidierungsreaktionsprodukt und der Bodenstrom zusätzlich Wasser umfassen.

7. Verfahren nach Anspruch 1, wobei der Oberstrom zu einer zweiten Fraktioniervorrichtung geführt wird, wobei der Oberstrom einer Destillation unterworfen wird, derartig daß ein zweiter Oberstrom, umfassend Propylen, und ein zweiter Unterstrom, umfassend Propylenoxid und hauptsächlich frei von Propylen, separat von der zweiten Frak-

tioniervorrichtung zurückgezogen werden.

8. Verfahren nach Anspruch 7, wobei der zweite Unterstrom zu einer extraktiven Destillationskolonne geführt wird, wobei der zweite Unterstrom einer extraktiven Destillation mit einem extraktiven Solvens unterworfen wird, derartig daß ein dritter Oberstrom im wesentlichen bestehend aus Propylenoxid und einer dritter Unterstrom umfassend das extraktive Solvens und die in dem zweiten Bodenstrom vorhandenen Unreinheiten separat von der extraktiven Destillationskolonne zurückgezogen werden.

9. Verfahren nach Anspruch 8, wobei das extraktive Solvens aus der Gruppe, bestehend aus Propylenglycol und Octan, gewählt ist.

10. Verfahren zum Reinigen von in einem Epoxidierungsprozeß produziertem Propylenoxid, wobei Methanol als ein Solvens, Hydrogenperoxid als ein Oxidans und ein Titan enthaltender Zeolith als Katalysator verwendet wird, umfassend die Schritte von:

a. Führen eines rohen Epoxidierungsreaktionsprodukts umfassend 2 bis 10 Gewichtsprozent Propylenoxid, 60 bis 85 Gewichtsprozent Methanol, 10 bis 25 Gewichtsprozent Wasser und 0,01 bis 0,1 Gewichtsprozent Acetaldehyde zu einer ersten Fraktioniervorrichtung mit 20 bis 60 theoretischen Dampf/Flüssigkeitskontaktschritten;

b. Unterwerfen des rohen Epoxidierungsreaktionsprodukts einer Fraktionierdestillation innerhalb der Fraktioniervorrichtung mit einem Oberdruck von 15 bis 50 psia, einer Bodentemperatur von 80°C bis 110°C und einem Rückfluß/Destillatverhältnis von 10:1 bis 30:1.

c. Zurückziehen von der Fraktioniervorrichtung eines Oberstroms, umfassend Popylenoxid und zumindest 2 Gewichtsprozentsatz Methanol; und

d. Zurückziehen von der Fraktioniervorrichtung eines Unterstroms umfassend Methanol, Wasser und zumindest 99% von dem in dem rohen Epoxidierungsreaktionsprodukt vorhandenen Acetaldehyde.

11. Verfahren nach Anspruch 10, wobei der Oberdruck 25 bis 40 psia (172,4 bis 275,8 kPa), die Bodentemperatur von 93°C bis 104°C, und das Rückfluß/Destillatverhältnis von 15:1 bis 25:1 ist.

12. Verfahren nach Anspruch 11, wobei der Oberstrom zumindest 4 Gewichtsprozent Methanol umfaßt.

13. Verfahren nach Anspruch 12, wobei der Bodenstrom zumindest 99% von dem orginal in dem rohen Expoxidierungsreaktionsprodukt vorhandenen Wasser enthält.

Revendications

1. Procédé de purification d'un produit brut de réaction d'époxydation comprenant 2 à 10 % en masse d'oxyde de propylène, 60 à 85 % en masse de méthanol, 10 à 25 % en masse d'eau et 0,01 à 0,1 % en masse d'acétaldéhyde comprenant les étapes de

(a) introduction du produit brut de réaction d'époxydation dans une colonne de fractionnement ayant de 20 à 60 étages de contact gaz-liquide théoriques ;
(b) soumission du produit brut de réaction d'époxydation à une distillation fractionnée dans la colonne de fractionnement à une pression de tête de 15 à 50 psia (103,4 à 344,7 kPa), une température de pied de 80°C à 110°C et un rapport reflux:distillat de 10:1 à 30:1 ;
(c) retrait de la colonne de fractionnement d'un courant de tête comprenant de l'oxyde de propylène et au moins 2 % en masse de méthanol ; et
(d) retrait de la colonne de fractionnement d'un premier courant de pied comprenant du méthanol, de l'eau et au moins 99 % de l'acétaldéhyde présent dans le produit brut de réaction d'époxydation.

2. Procédé selon la revendication 1 où ledit procédé d'époxydation utilise comme catalyseur une zéolite contenant du titane.

3. Procédé selon la revendication 1 où ledit procédé d'époxydation utilise du peroxyde d'hydrogène comme oxydant.

4. Procédé selon la revendication 1 où la concentration du méthanol dans le produit brut de réaction d'époxydation est de 60 à 80 % en masse.

5. Procédé selon la revendication 1 où le produit brut de réaction d'époxydation contient initialement du propylène et le produit brut de réaction d'époxydation est soumis avant l'étape (a) à une distillation initiale où une partie sensible du propylène est retirée.

6. Procédé selon la revendication 1 où le produit brut de réaction d'époxydation et le courant de pied comprennent en outre de l'eau.

7. Procédé selon la revendication 1 où le courant de tête est introduit dans une seconde colonne de fractionnement où le courant de tête est soumis à une distillation de sorte qu'un second courant de tête comprenant du propylène et un second courant de pied comprenant de l'oxyde de propylène et sensiblement exempt de propylène sont retirés séparément de la seconde colonne de fractionnement.

8. Procédé selon la revendication 7 où le second courant de pied est introduit dans une colonne de distillation extractive où le second courant de pied est soumis à une distillation extractive avec un solvant d'extraction de sorte qu'un troisième courant de tête consistant essentiellement en oxyde de propylène et un troisième courant de pied comprenant le solvant d'extraction et des impuretés présentes dans le second courant de pied sont retirés séparément de la colonne de distillation extractive.

9. Procédé selon la revendication 8 où le solvant d'extraction est choisi dans le groupe consistant en le propylène-glycol et l'octane.

10. Procédé de purification de l'oxyde de propylène produit dans un procédé d'époxydation où le méthanol est utilisé comme solvant, le peroxyde d'hydrogène est utilisé comme oxydant et une zéolite contenant du titane est utilisée comme catalyseur comprenant les étapes de

    (a) introduction d'un produit brut de réaction d'époxydation comprenant 2 à 10 % en masse d'oxyde de propylène, 60 à 85 % en masse de méthanol, 10 à 25 % en masse d'eau et 0,01 à 0,1 % en masse d'acétaldéhyde dans une première colonne de fractionnement ayant de 20 à 60 étages de contact gaz-liquide théoriques ;
    (b) soumission du produit brut de réaction d'époxydation à une distillation fractionnée dans la colonne de fractionnement à une pression de tête de 15 à 50 psia, une température de pied de 80°C à 110°C et un rapport reflux:distillat de 10:1 à 30:1 ;
    (c) retrait de la colonne de fractionnement d'un courant de tête comprenant de l'oxyde de propylène et au moins 2 % en masse de méthanol ;
    (d) retrait de la colonne de fractionnement d'un courant de pied comprenant du méthanol, de l'eau et au moins 99 % de l'acétaldéhyde présent dans le produit brut de réaction d'époxydation.

11. Procédé selon la revendication 10 où la pression de tête est de 25 à 40 psia (172,4 à 275,8 kPa), la température de pied est de 93°C à 104°C et le rapport reflux :distillat est de 15:1 à 25:1.

12. Procédé selon la revendication 11 où le courant de tête comprend au moins 4 % en masse de méthanol.

13. Procédé selon la revendication 12 où le courant de pied contient au moins 99 % de l'eau présente initialement dans le produit brut de réaction d'époxydation.

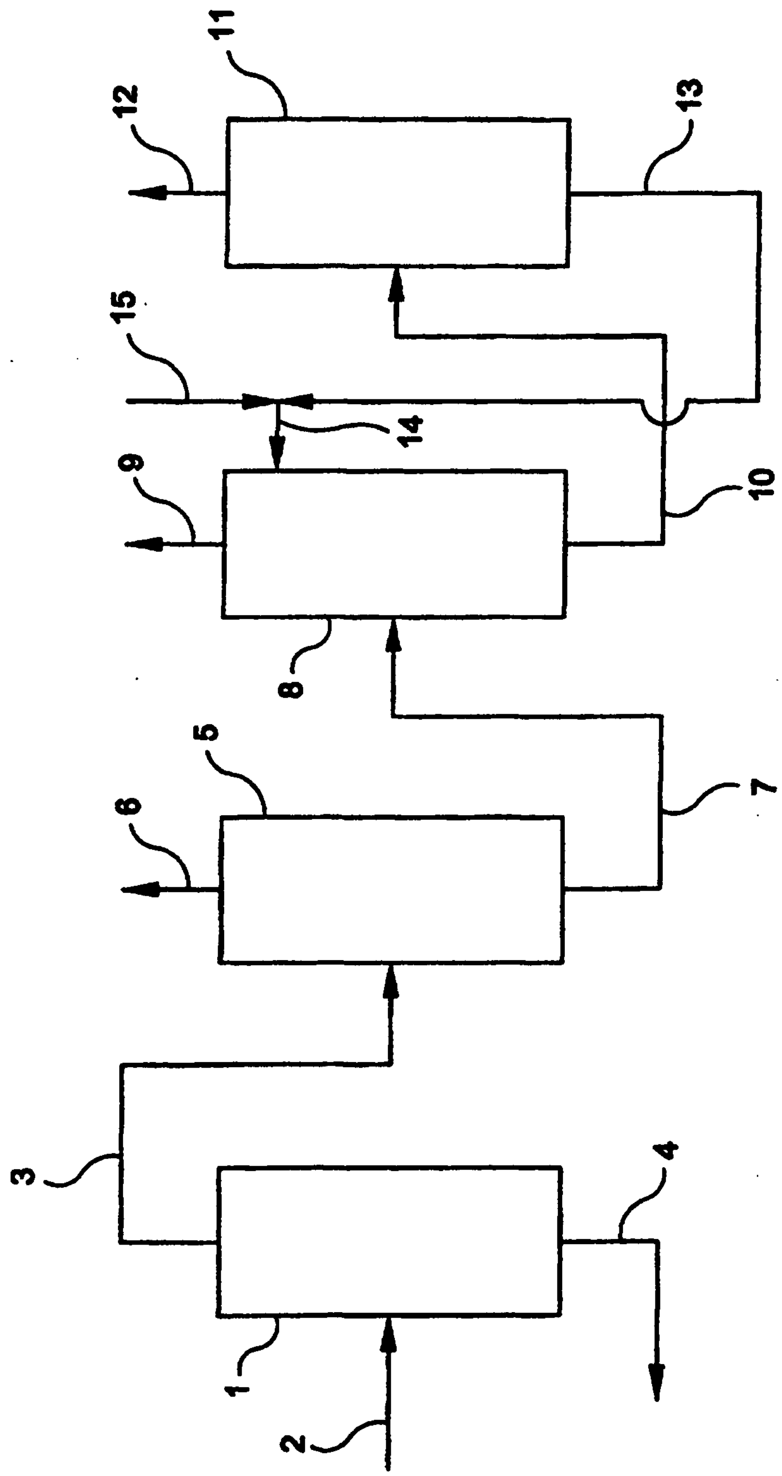

FIG-1